NANO VISION
An International Open Free Access, Peer Reviewed Research Journal
www.nano-journal.org

ISSN 2231-2579 (Print)
ISSN 2319-7633 (Online)
Abbr: Nano Vision.
2014, Vol.4(1): Pg.17- 20

Simple Thermal CVD Method for Synthesis of Nanoscopic Entities of Carbon

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(Received on: February 14, 2014)

ABSTRACT

Simple CVD method can be utilized for the preparation of carbon nanotubes, carbon nanoflowers and carbon nanosponge with slight modification in the process for each entity. Here we report cost-effective, clean methods without the use of an external catalyst.

Keywords: Carbon nanotubes, nano flowers, CNT Sponge, SEM, TEM, FT-IR, AFM.

1. INTRODUCTION

Carbon nanostructures have gained more importance recently due their potential that can be exploited for electronic application (1), in biotechnology (2) and biomedicine (3).However the lack of cost-effective method for synthesis limits the application of these unique materials. Chemical vapor deposition (CVD) method represents the most promising method for large scale production (4). Here the detailed structural studies of the nanomaterials with graphitic features obtained by slightly modifying the procedure for each synthesis reveals that they contain multiwalled carbon nanotube (MWCNT)(5), carbon nanoflowers (6) and carbon nanosponge(7). Here the multiwalled nano tubes were synthesized directly on stainless steel plate(SS 304)by thermal CVD method without the external addition of a catalyst precursor. Carbon nanoflowers were synthesized on the same substrate by coating it with a Fe catalyst. CNT Sponge is obtained by CVD process using ferrocene and 1, 2 dichloro benzene as the catalyst precursor and carbon source, respectively.

2. EXPERIMENTAL

2.1 Synthesis of CNT

Substrate (SS 304) was cleaned by sonicating in acetone for 30 min. followed by itching in HCl for 10 min. and was placed

directly in the Recrystalised Alumina tube(50 OD x 40ID x 750 long) housed in a resistive furnace. Substrate was heated at 850^0C for 30 min. in N_2 atmosphere (at a flow rate of 592 SCCM) followed by a flow of Acetylene (carbon source) at a flow rate of 45 SCCM at 700^0C for 5 min. A growth period of 30 min. was given at 700^0C in N_2 (592 SCCM) followed by cool down to room temperature.CNT deposited on the substrate was characterized with SEM and TEM.

2.2 Synthesis of Carbon nanoflowers

Furnace and the methodology used for the synthesis of this nanoform of carbon was also same as that used for CNT synthesis except the fact that the substrate was uniformly coated with a Fe catalyst by dipping into a weak solution of $FeCl_2$ and dried by nitrogen flow. The surface patterns of the sample deposited on the substrate were characterized by SEM and AFM analysis.

2.3 Synthesis of CNT Sponge

CNT Sponges were synthesized by CVD process using 1,2 dichlorobenzene as a source of carbon and ferrocene as the catalyst precursor. A solution of ferrocene in 1, 2 dichloro benzene (0.06 gml^{-1}) was injected to the Al_2O_3 tube in the furnace by a syringe pump in a rate of 7.8 ml/hr. A quartz plate (2×1 inch) sonicated with HF for 2 min. followed by washing with acetone and used as a substrate. T he reaction temperature was set at 860^0C. A mixture of Argon and Hydrogen (carrier gases) are allowed to flow at a rate of 2000 sccm and 300 sccm respectively for a period of 4 hrs. The sponge- like material obtained is subjected to detailed structural investigation. .Chemical stability of the mixture is studied by dipping it in various solvents for 48 hrs and then taking FT-IR.

3. RESULTS AND DISCUSSIONS

First, the surface pattern of these materials was visualized by using scanning electron microscopy (SEM). Further investigations were done with transmission electron microscopy (TEM) and FT-IR. SEM micrographs shows the formation of different nanoscopic entities which can be confirmed with TEM image.

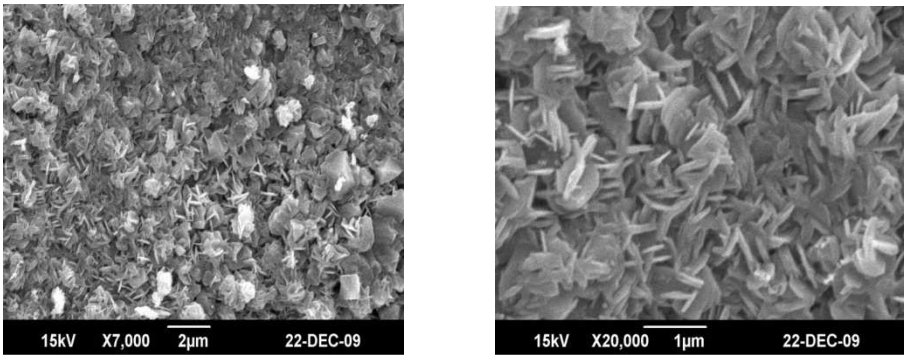

Fig 1.(a) Low-magnification SEM images of carbon nanoflowers.

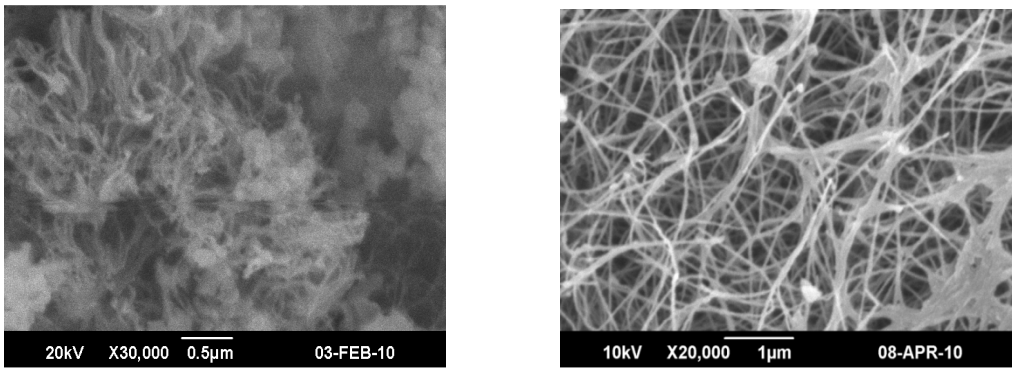

Fig 2. Low-magnification SEM images of (a) carbon nanotubes (b) carbon nano sponge.

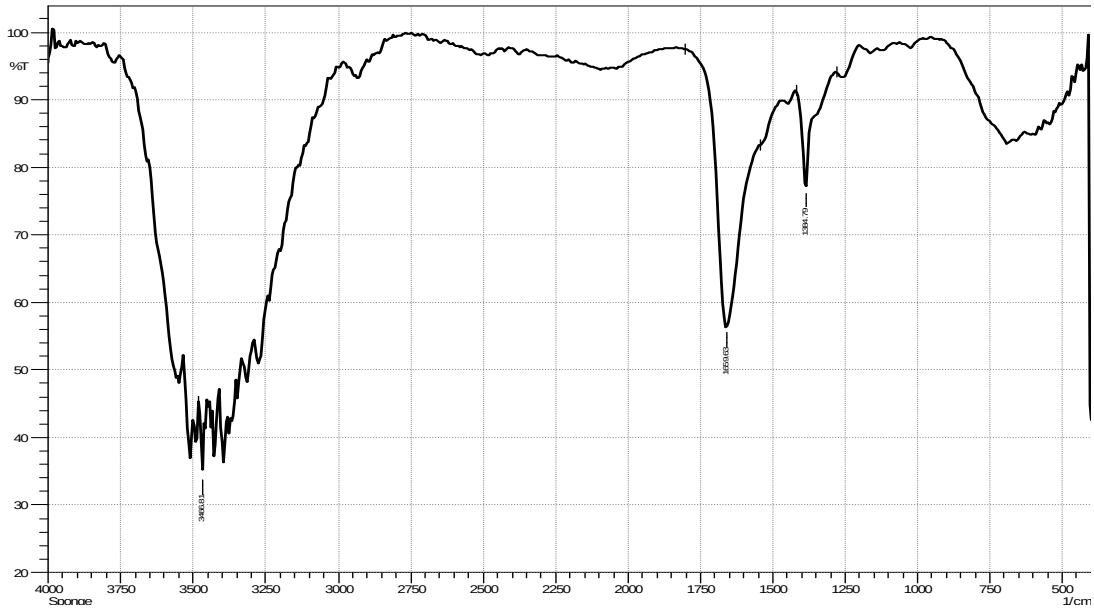

Fig 3.FT-IR spectra of amino-CNT Sponge

ACKNOWLEDGMENT

I acknowledge Dr. Roice Micheal and Dr. C.P Vinod, Cardiff University, Cardiff, UK for their support and guidance.

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